REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED** *(From - To)*
24 September 2015	Briefing Charts	20 August 2015 – 24 September 2015

4. TITLE AND SUBTITLE
Air Force Dynamic Mechanical Analysis of NATO Round Robin Propellant Testing for Development of AOP-4717

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
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5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER
Q16H

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Air Force Research Laboratory (AFMC)
AFRL/RQRP
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8. PERFORMING ORGANIZATION REPORT NO.

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Air Force Research Laboratory (AFMC)
AFRL/RQR
5 Pollux Drive
Edwards AFB, CA 93524-7048

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
AFRL-RQ-ED-VG-2015-347

12. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for public release; distribution unlimited

13. SUPPLEMENTARY NOTES
For presentation at NATO A/C 326 Working Group Meeting; U.S. Army Research, Development and Engineering Command, Huntsville, Alabama; 22-24 Sep 2015
PA Case Number: #15565; Clearance Date: 9/18/2015

14. ABSTRACT
Viewgraph/Briefing Charts

15. SUBJECT TERMS
N/A

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
					T. Miller
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**	SAR	24	**19b. TELEPHONE NO** *(include area code)*
Unclassified	**Unclassified**	**Unclassified**			N/A

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. 239.18

Air Force Dynamic Mechanical Analysis of NATO Round Robin Propellant Testing for Development of AOP-4717

23 Sep 2015

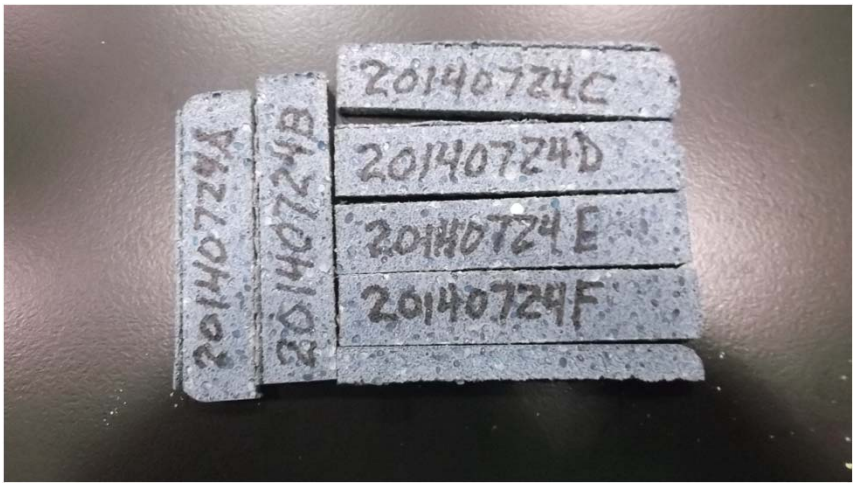
Tim Miller
U.S. Air Force Research Lab
Propulsion Division

Introduction

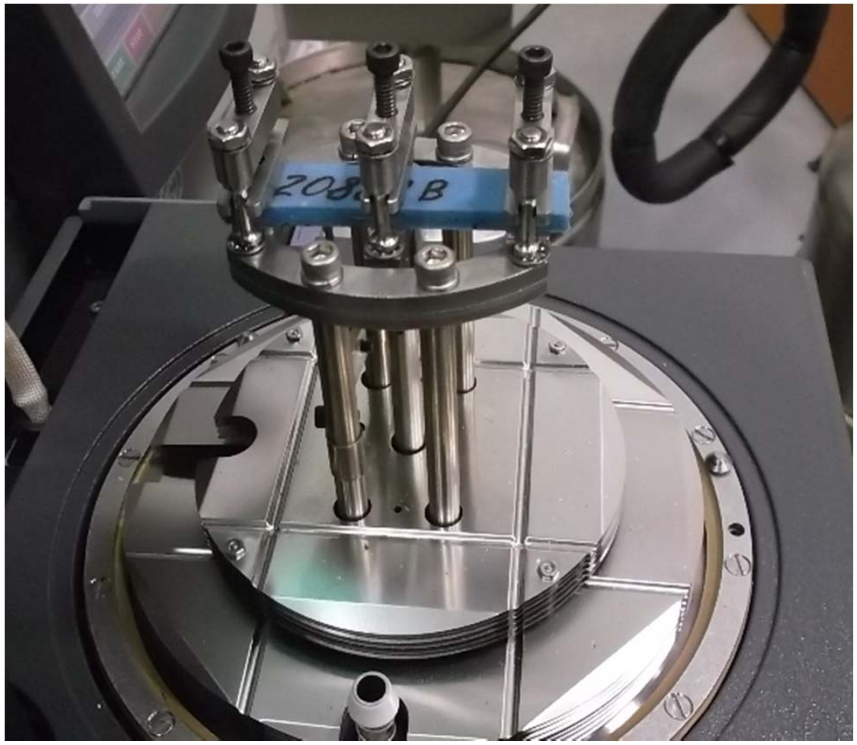
Specimen name	Test type	Relative Humidity	Temperature (deg C)
20140724A	Strain sweep	53.5	18.1
20140724B			
20140724C			
20140724D	Frequency sweep	73.3	20.6
20140724E		61.8	21.5
20140724F		52.8	19.6

Specimen name	Width (mm)	Thickness (mm)	Length (mm)
20140724A	12.65	3.99	56.19
20140724B	12.79	4.11	56.37
20140724C	12.75	4.10	64.20
20140724D	12.19	4.12	63.57
20140724E	12.63	4.12	63.95
20140724F	12.98	4.16	63.73

Introduction



Introduction



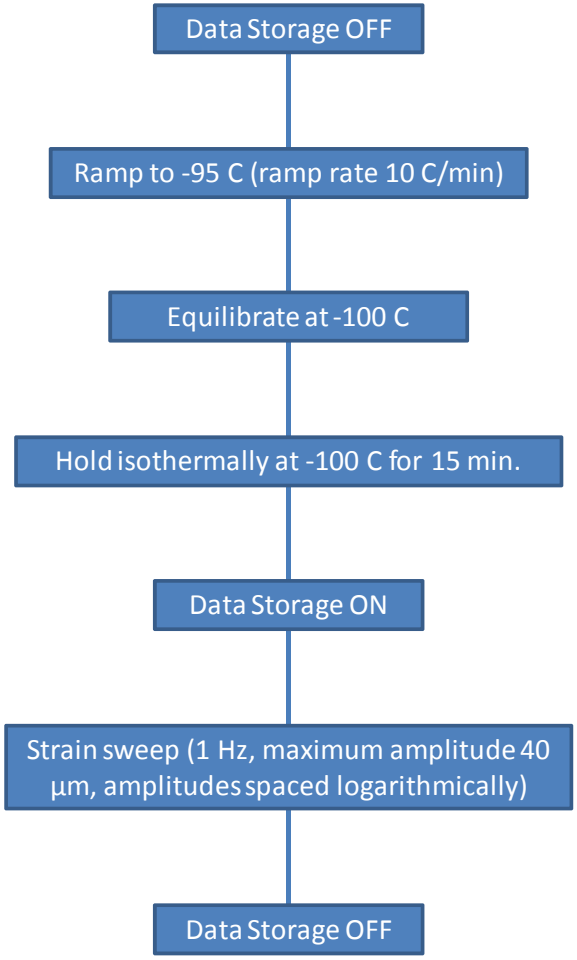
Strain Sweep Tests

Specimen name	Test temperature (C)
20140724A	-100
20140724B	21
20140724C	70

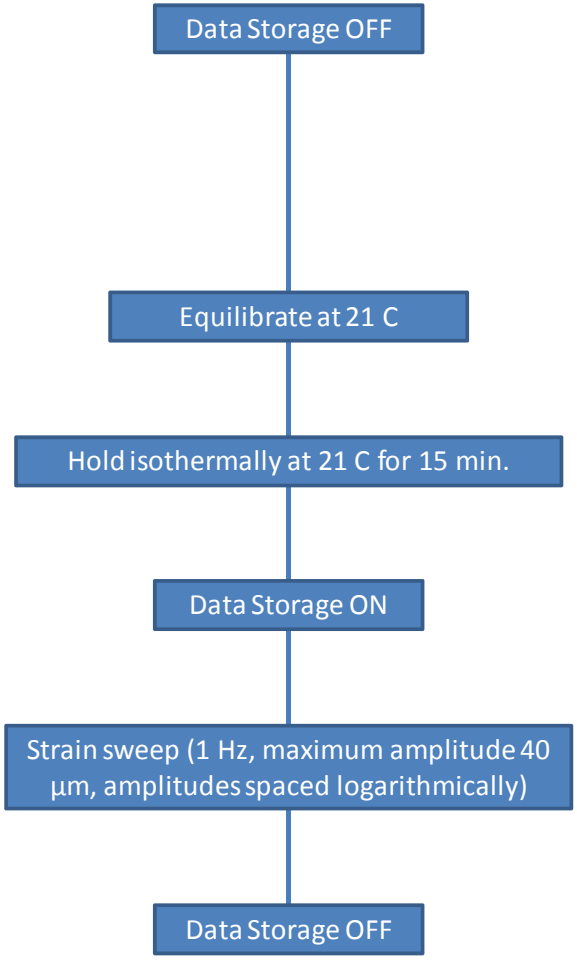
One test per test temperature. Amplitude varied, frequency always 1 Hz

Strain Sweep Tests

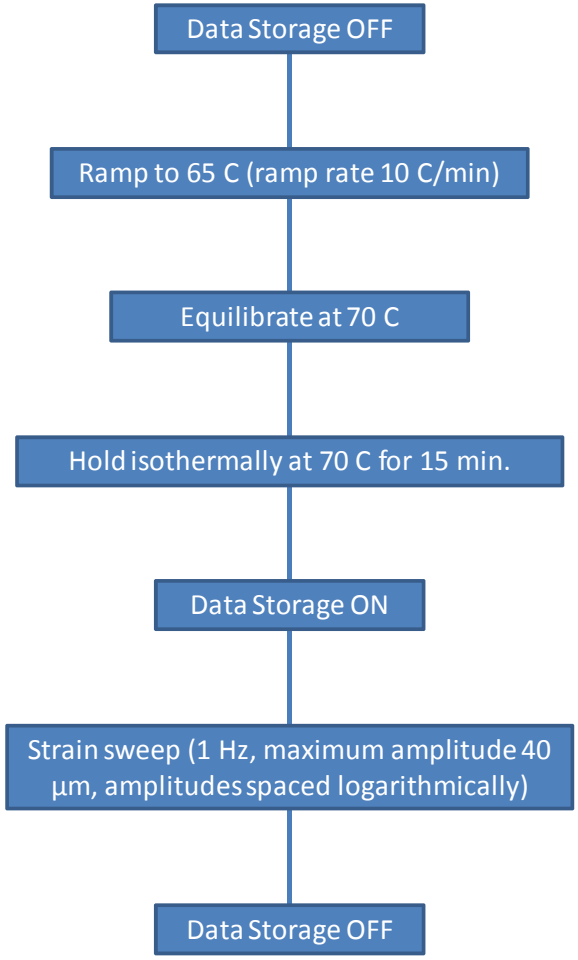
Strain sweep block diagram for -100 C



Strain sweep block diagram for 21 C

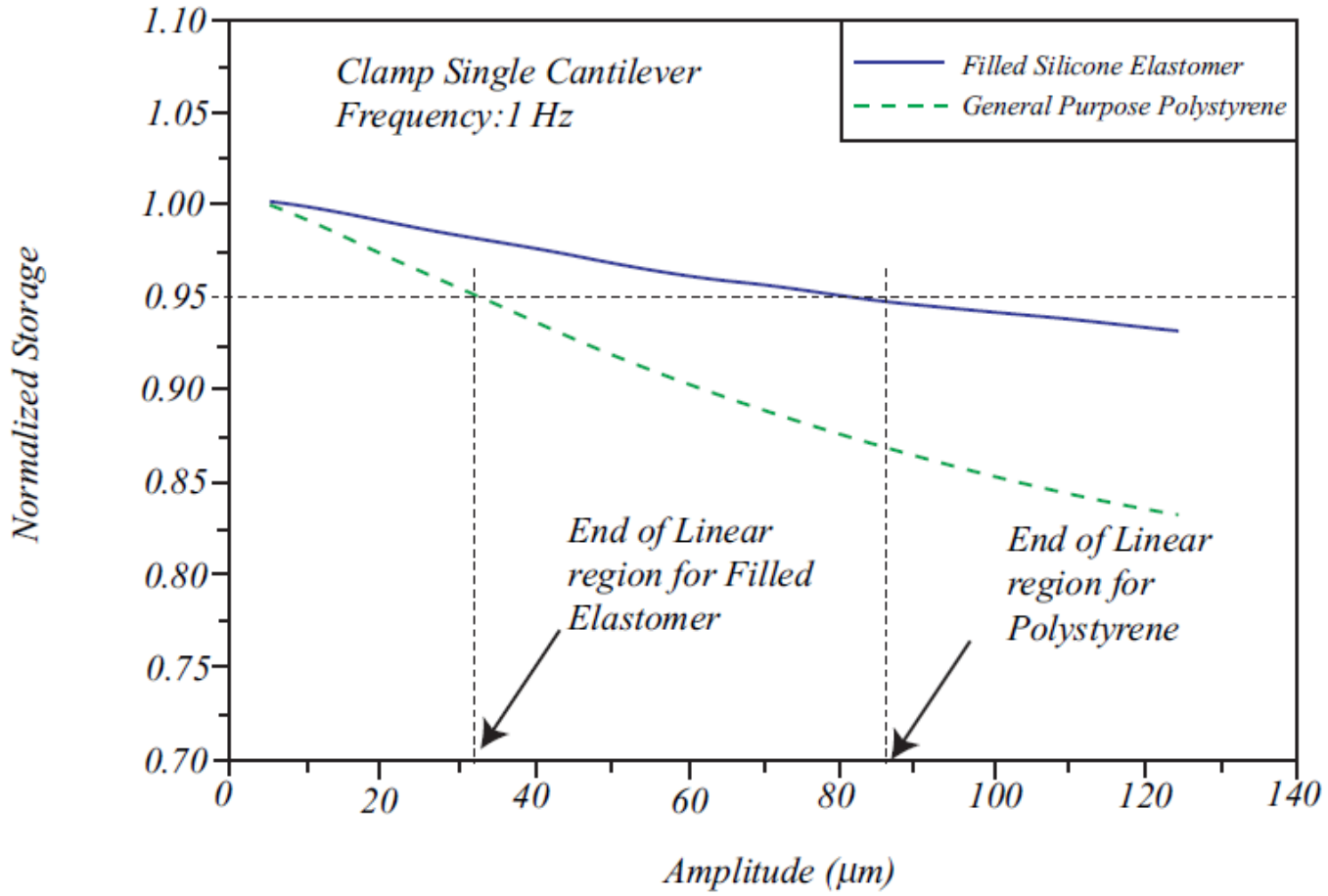


Strain sweep block diagram for 70 C

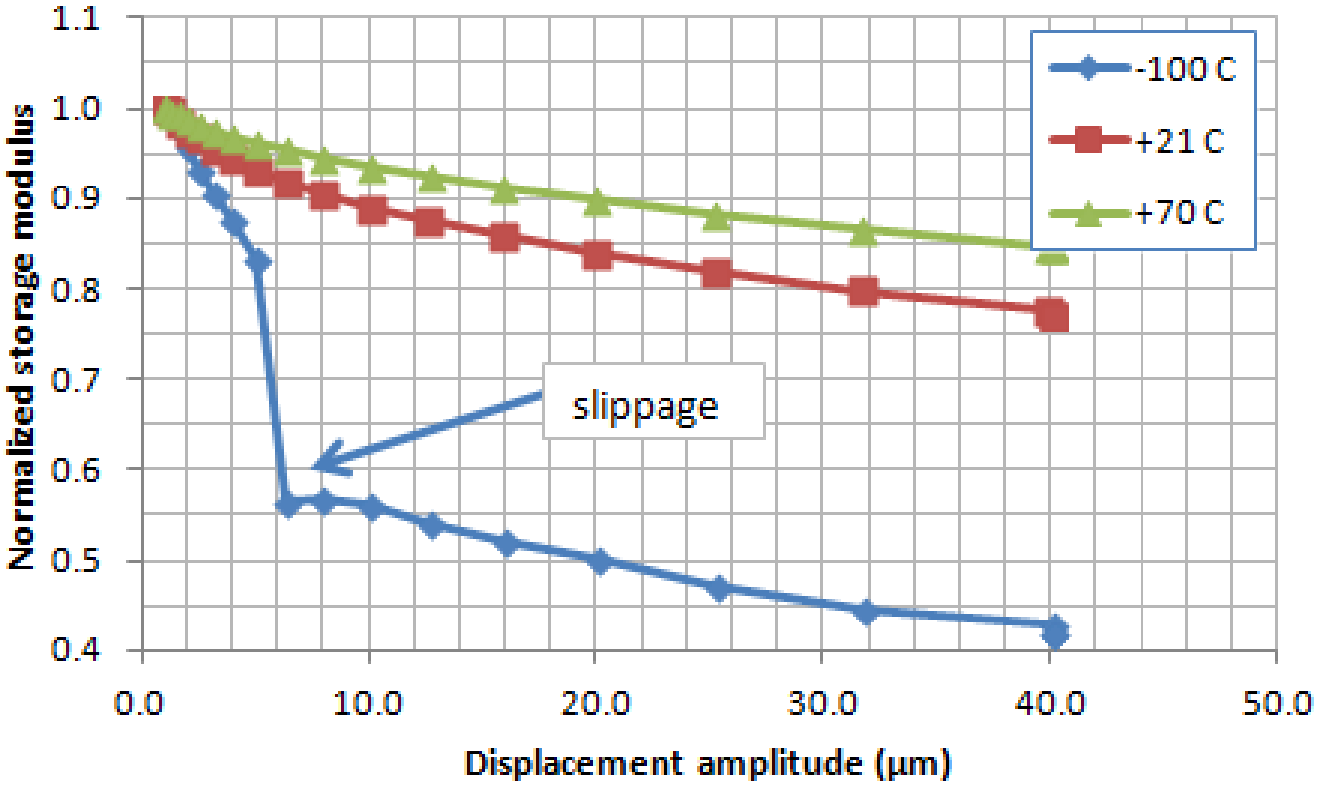


Strain Sweep Tests

Comparison of Normalized Storage Modulus for a Filled Silicone Elastomer and a General Purpose Polystyrene



Strain Sweep Tests

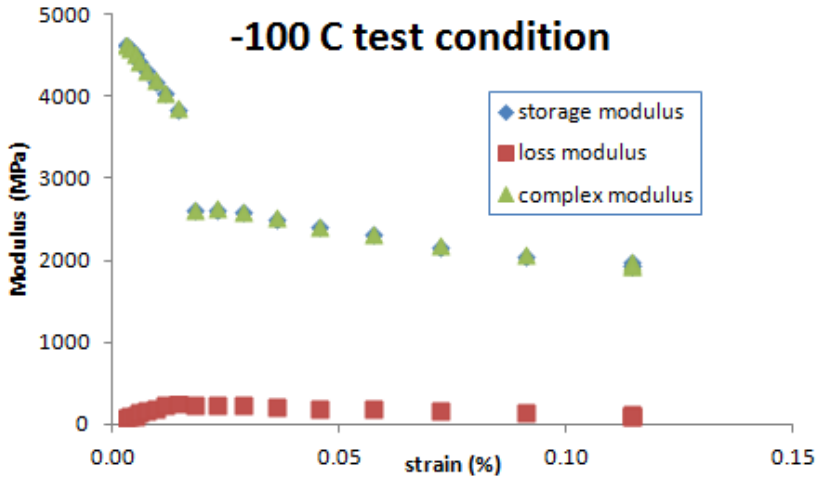


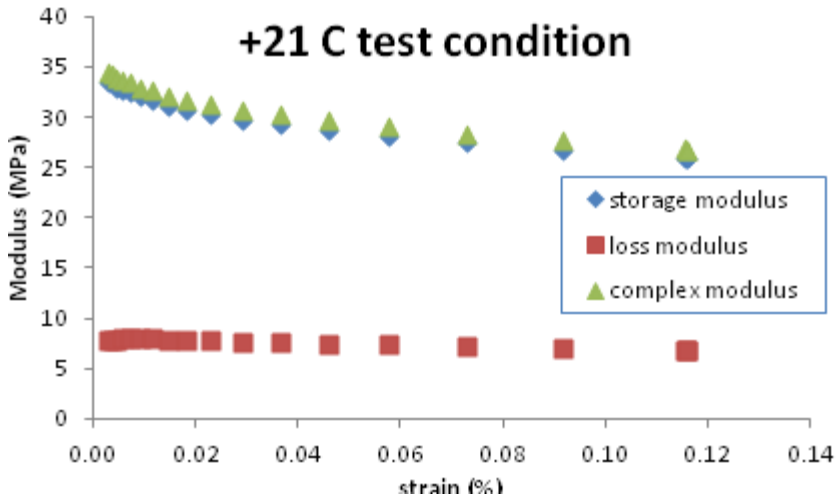
Strain Sweep Tests

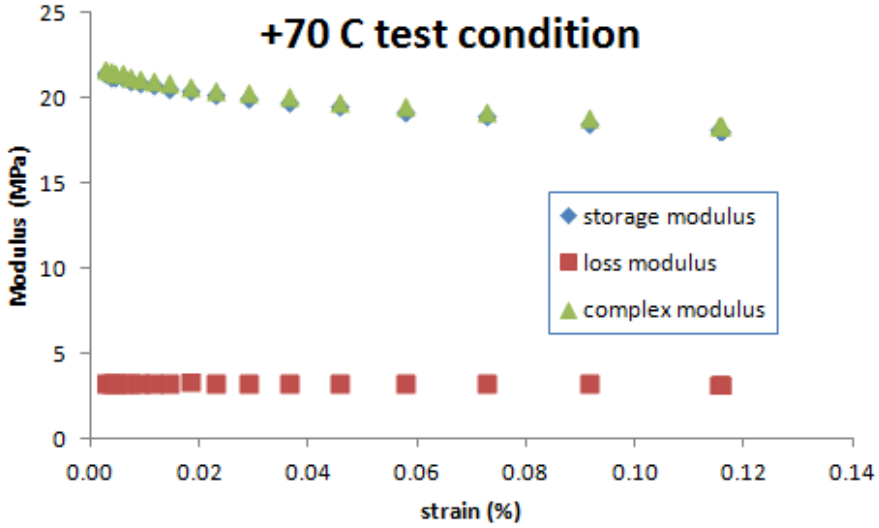
Test condition (C)	Upper limit for amplitude for linear viscoelasticity (μm)
-100	2.15
+21	3.58
+70	7.06

This comes from the data on the previous slide. The frequency sweep tests all had the same amplitude, 2.10 μm, so regardless of temperature linear viscoelasticity was ensured.

Strain Sweep Tests

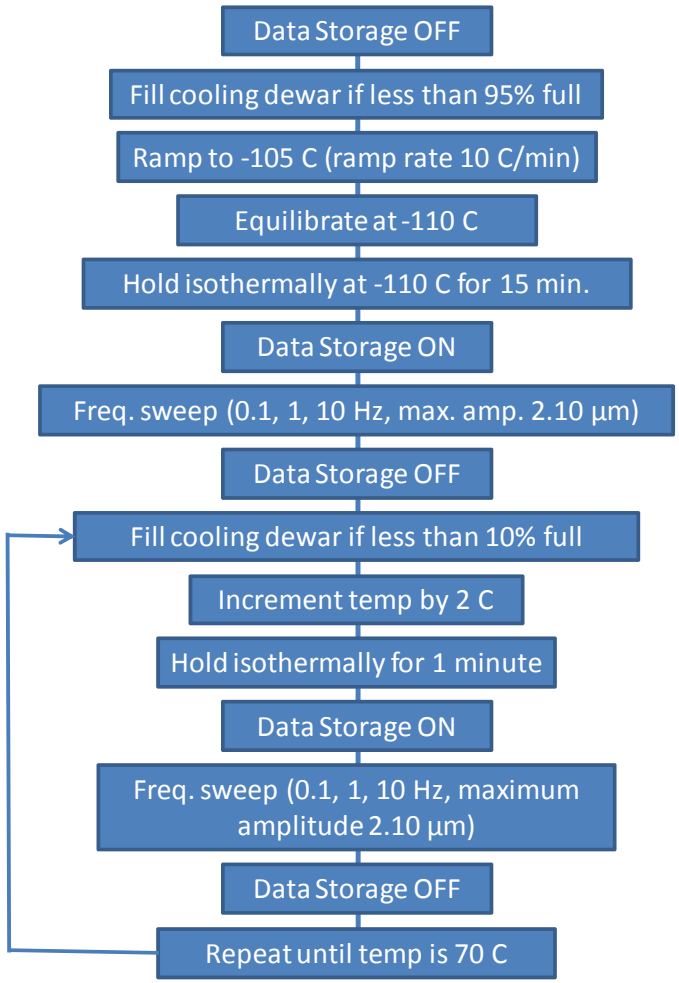




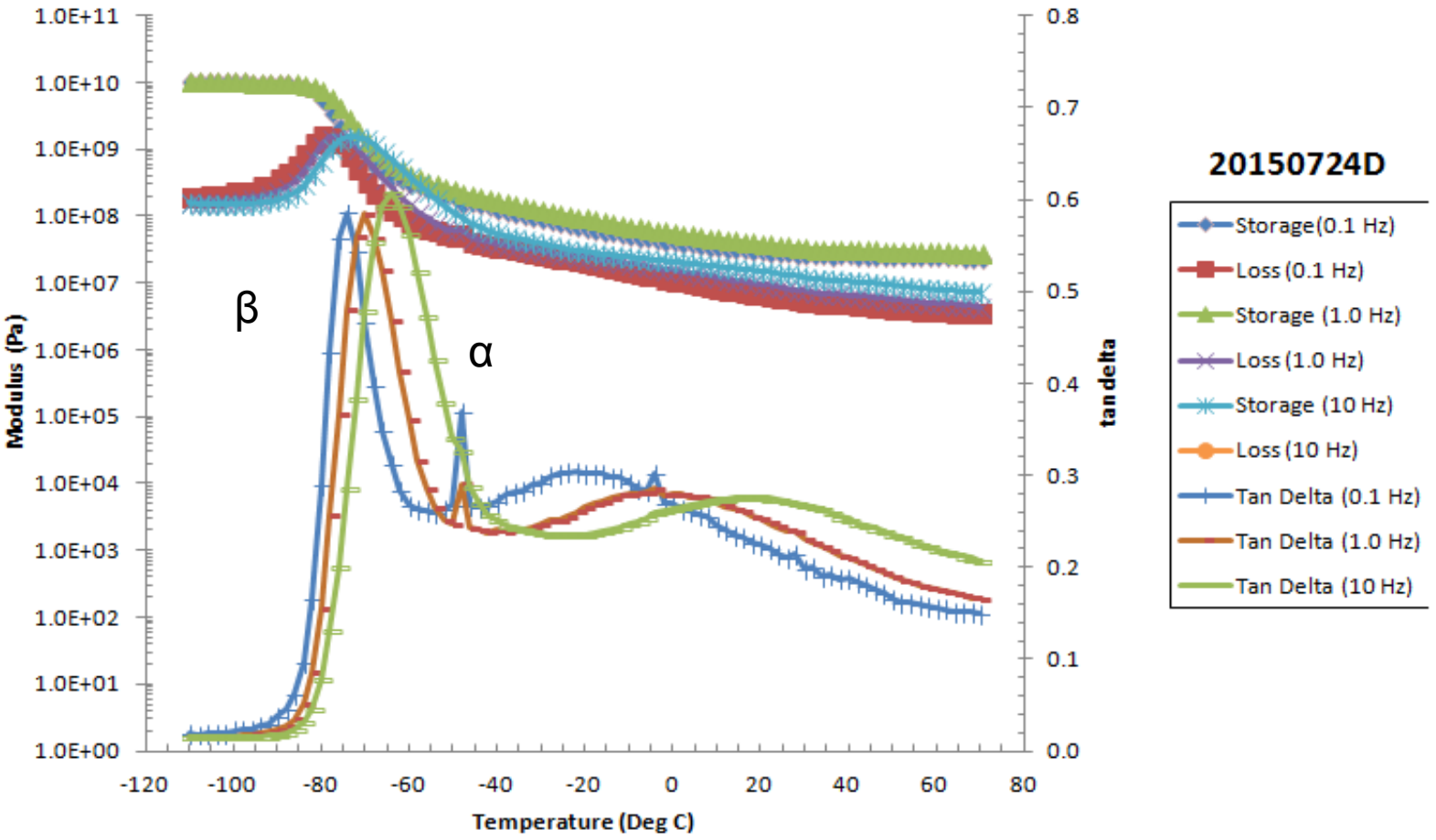


Frequency Sweep Tests

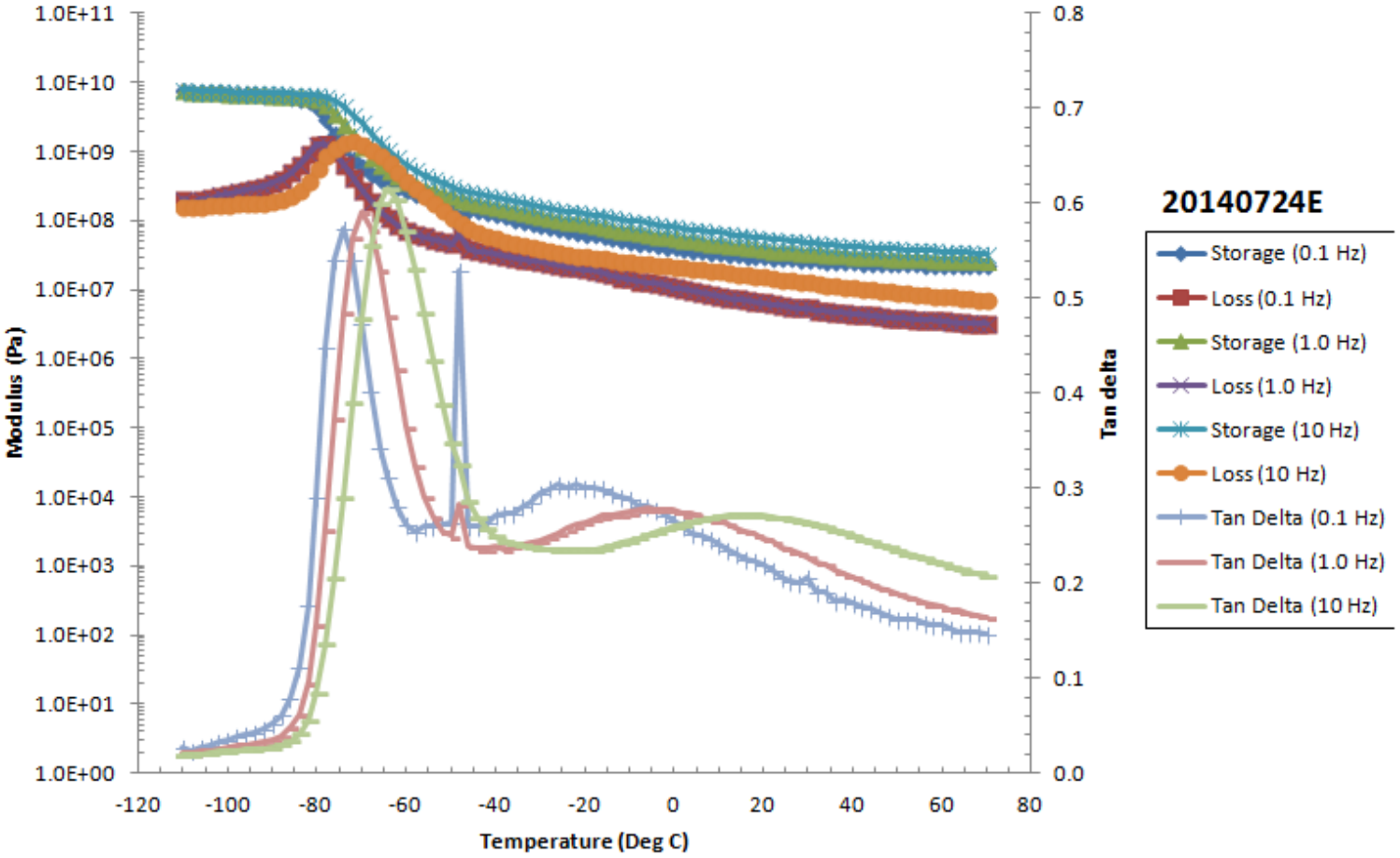
Frequency sweep block diagram



Frequency Sweep Tests



Frequency Sweep Tests



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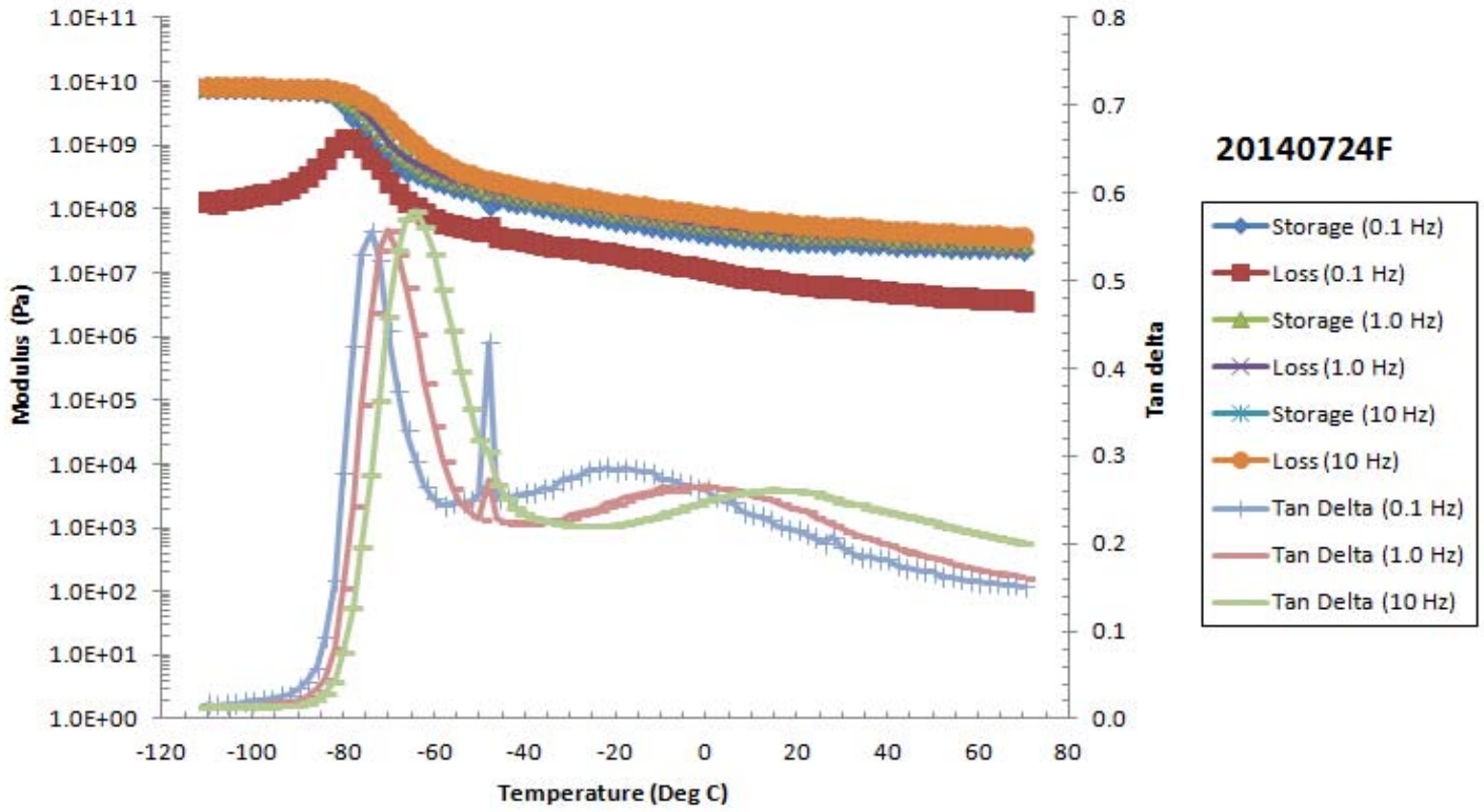
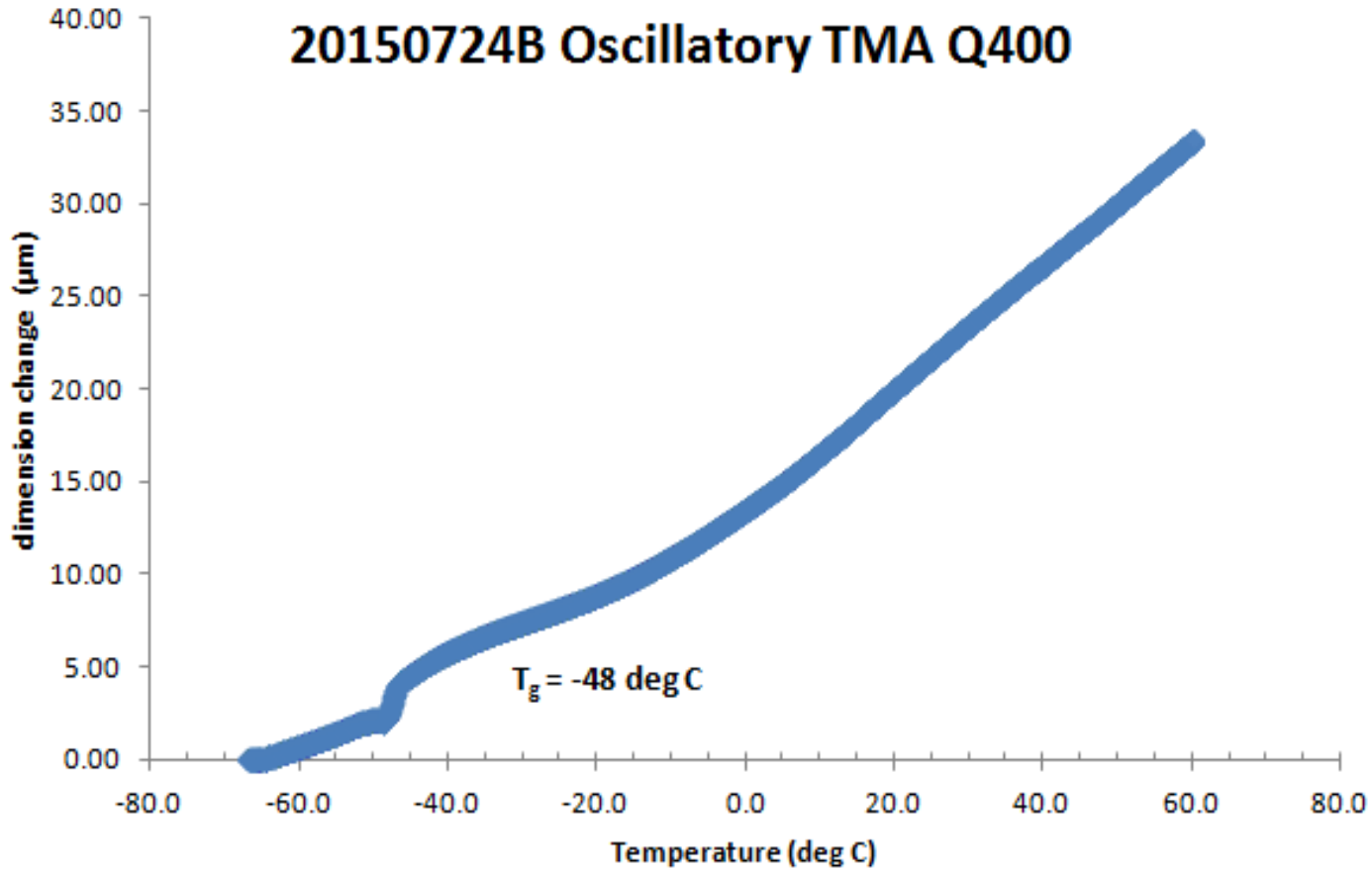


Frequency Sweep Tests

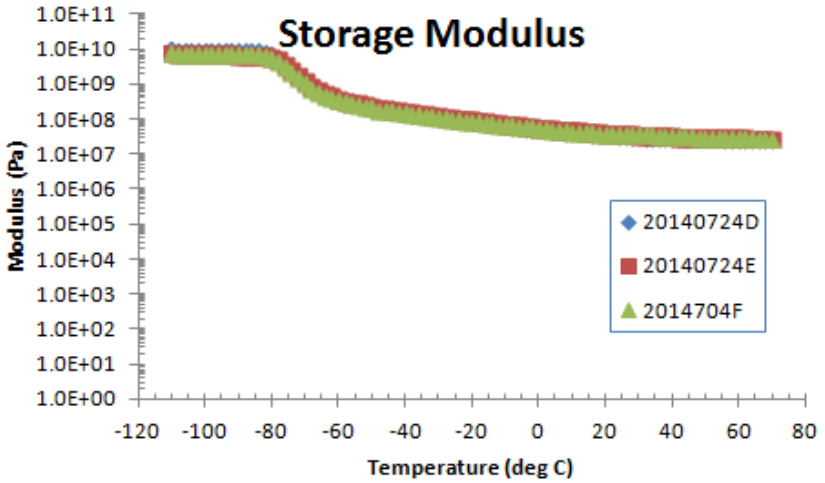
Specimen name	α peaks (deg C)			β peaks (deg C)		
	0.1 Hz	1.0 Hz	10 Hz	0.1 Hz	1.0 Hz	10 Hz
20140724D	-48.0	-48.0	-48.0	-74.0	-70.0	-64.0
20140724E	-48.0	-48.0	-48.0	-74.0	-70.0	-64.0
20140724F	-48.0	-48.0	-46.0	-74.0	-70.0	-64.0

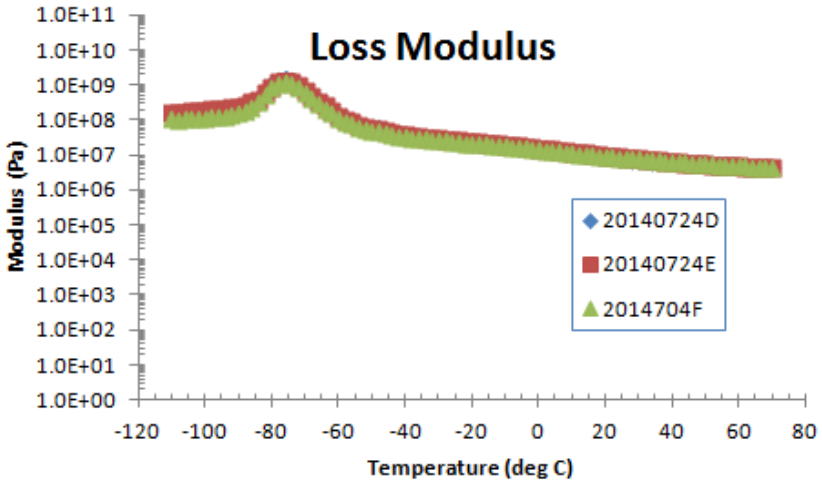
Very consistent peak values for temperature among specimens. T_g not affected by frequency, apparently.

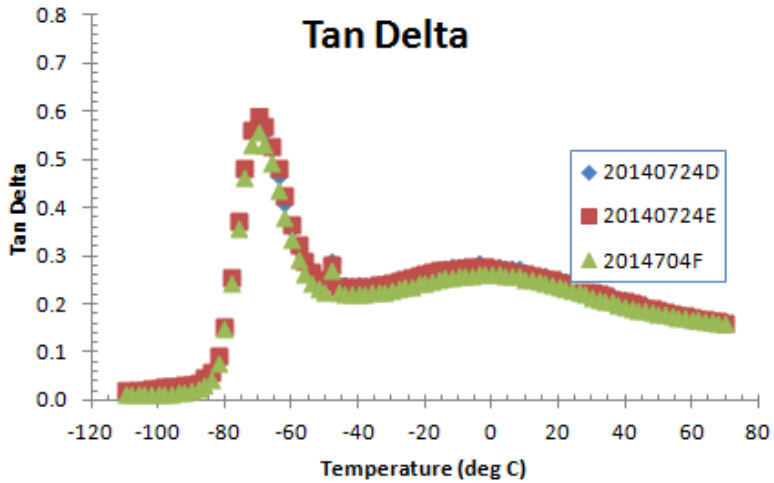
Frequency Sweep Tests



Frequency Sweep Tests



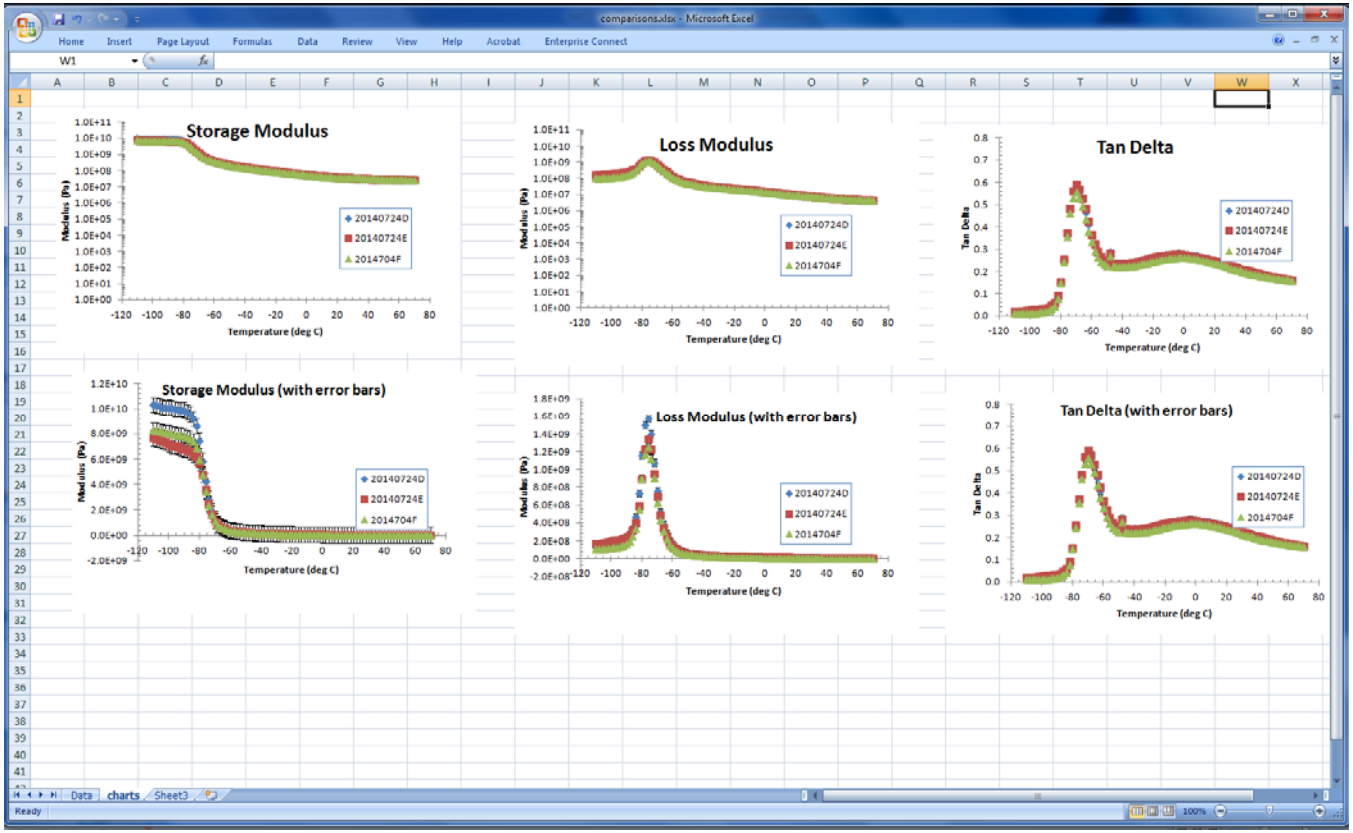




Freq. Sweep Tests (Bonus Information)

$$s_p^2 = \frac{\sum_{i=1}^{k}(n_i - 1)s_i^2}{\sum_{i=1}^{k}(n_i - 1)} = \frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2 + \cdots + (n_k - 1)s_k^2}{n_1 + n_2 + \cdots + n_k - k}$$

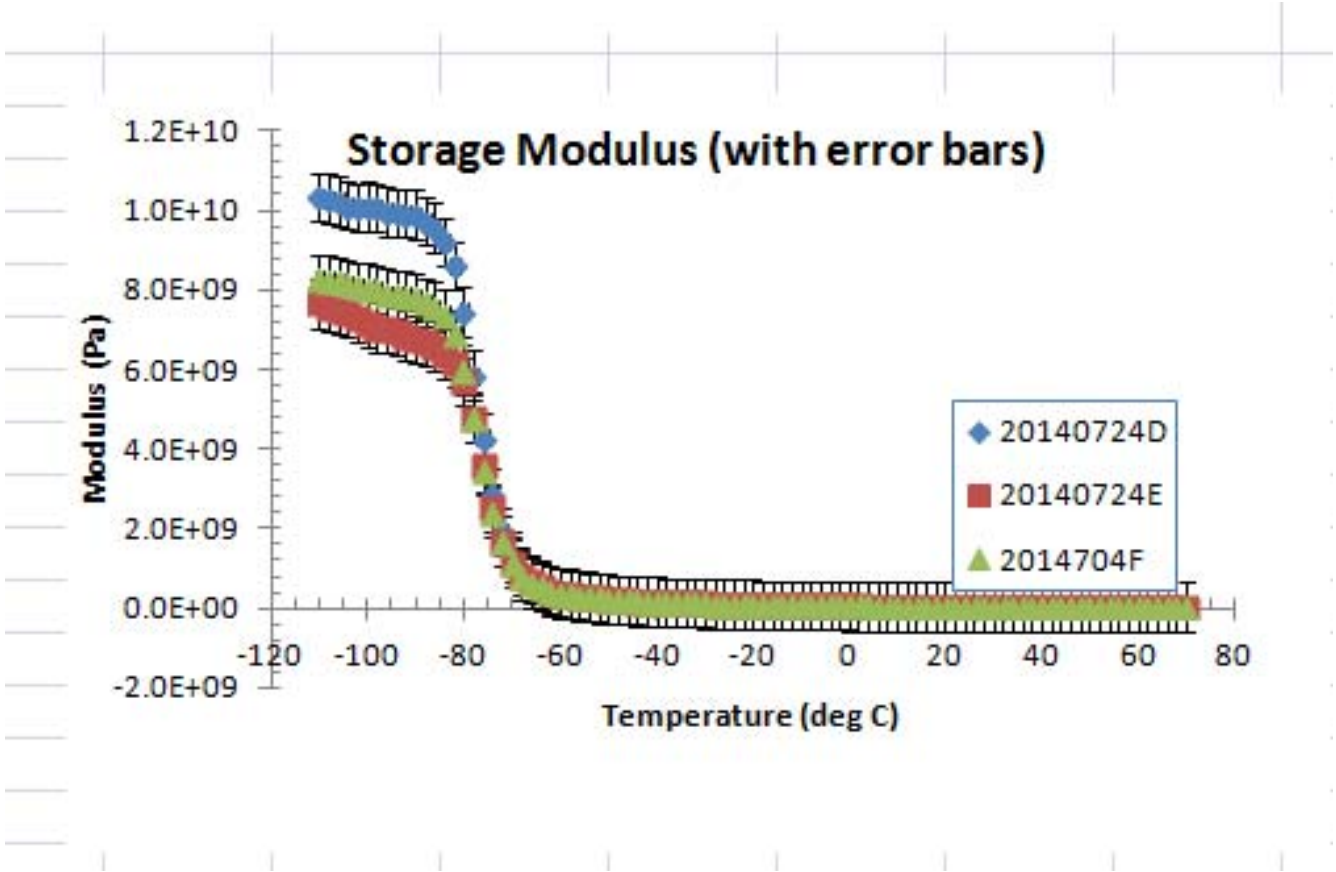
wikipedia “pooled variances” (for more detail, see book by John Mandel on experiments and statistics)



Freq. Sweep Tests (Bonus Information)

	20140724D (0.1 Hz)			20140724E (0.1 Hz)			20140724F (0.1 Hz)			20140724D (1.0 Hz)			201
Nominal Temperature (deg C)	Storage Modulus (Pa)	Loss Modulus (Pa)	Tan Delta	Storage Modulus (Pa)	Loss Modulus (Pa)	Tan Delta	Storage Modulus (Pa)	Loss Modulus (Pa)	Tan Delta	Storage Modulus (Pa)	Loss Modulus (Pa)	Tan Delta	Storage Modulus (Pa)
-110	1.020E+10	1.883E+08	0.0185	7.554E+09	2.053E+08	0.0272	8.142E+09	1.334E+08	0.0164	1.033E+10	1.589E+08	0.0154	7.664E+09
-108	1.014E+10	1.843E+08	0.0182	7.470E+09	1.777E+08	0.0238	8.134E+09	1.235E+08	0.0152	1.029E+10	1.597E+08	0.0155	7.592E+09
-106	1.008E+10	1.976E+08	0.0196	7.356E+09	2.007E+08	0.0273	8.086E+09	1.401E+08	0.0173	1.025E+10	1.636E+08	0.0160	7.479E+09
-104	9.967E+09	1.874E+08	0.0188	7.286E+09	2.104E+08	0.0289	8.024E+09	1.374E+08	0.0171	1.013E+10	1.629E+08	0.0161	7.416E+09
-102	9.914E+09	1.962E+08	0.0198	7.185E+09	2.336E+08	0.0325	7.956E+09	1.474E+08	0.0185	1.008E+10	1.636E+08	0.0162	7.329E+09
-100	9.913E+09	2.222E+08	0.0224	7.037E+09	2.406E+08	0.0342	7.911E+09	1.581E+08	0.0200	1.010E+10	1.626E+08	0.0161	7.179E+09
-98	9.889E+09	2.341E+08	0.0237	6.934E+09	2.639E+08	0.0381	7.868E+09	1.710E+08	0.0217	1.008E+10	1.786E+08	0.0177	7.067E+09
-96	9.798E+09	2.299E+08	0.0235	6.845E+09	2.802E+08	0.0409	7.781E+09	1.739E+08	0.0223	9.982E+09	1.805E+08	0.0181	7.014E+09
-94	9.737E+09	2.740E+08	0.0281	6.779E+09	2.898E+08	0.0427	7.711E+09	2.019E+08	0.0262	9.943E+09	1.908E+08	0.0192	6.945E+09
-92	9.695E+09	2.853E+08	0.0294	6.648E+09	3.087E+08	0.0464	7.677E+09	2.087E+08	0.0272	9.936E+09	2.076E+08	0.0209	6.842E+09
-90	9.608E+09	3.672E+08	0.0382	6.557E+09	3.461E+08	0.0528	7.602E+09	2.671E+08	0.0351	9.889E+09	2.358E+08	0.0238	6.767E+09
-88	9.409E+09	4.346E+08	0.0462	6.433E+09	3.912E+08	0.0608	7.422E+09	3.163E+08	0.0426	9.722E+09	2.654E+08	0.0273	6.706E+09
-86	9.090E+09	5.597E+08	0.0616	6.250E+09	4.924E+08	0.0788	7.214E+09	4.219E+08	0.0585	9.555E+09	3.264E+08	0.0342	6.601E+09
-84	8.457E+09	8.251E+08	0.0976	5.872E+09	6.529E+08	0.1112	6.827E+09	6.356E+08	0.0931	9.210E+09	4.694E+08	0.0510	6.396E+09
-82	7.386E+09	1.224E+09	0.1657	5.270E+09	9.337E+08	0.1772	5.930E+09	9.329E+08	0.1573	8.622E+09	7.330E+08	0.0850	6.148E+09
-80	5.433E+09	1.575E+09	0.2898	4.297E+09	1.248E+09	0.2904	4.439E+09	1.248E+09	0.2812	7.459E+09	1.160E+09	0.1555	5.711E+09
-78	3.459E+09	1.505E+09	0.4351	2.896E+09	1.298E+09	0.4481	2.914E+09	1.235E+09	0.4240	5.853E+09	1.504E+09	0.2569	4.771E+09
-76	2.114E+09	1.182E+09	0.5593	1.782E+09	9.642E+08	0.5411	1.769E+09	9.383E+08	0.5303	4.281E+09	1.573E+09	0.3674	3.564E+09
-74	1.325E+09	7.760E+08	0.5857	1.154E+09	6.621E+08	0.5736	1.139E+09	6.326E+08	0.5554	2.916E+09	1.399E+09	0.4800	2.517E+09
-72	9.019E+08	4.910E+08	0.5444	7.915E+08	4.282E+08	0.5410	8.183E+08	4.288E+08	0.5241	1.931E+09	1.080E+09	0.5591	1.680E+09
-70	6.626E+08	3.097E+08	0.4675	5.928E+08	2.803E+08	0.4728	5.992E+08	2.656E+08	0.4432	1.305E+09	7.624E+08	0.5842	1.171E+09
-68	5.284E+08	2.104E+08	0.3981	4.726E+08	1.904E+08	0.4029	4.738E+08	1.776E+08	0.3748	9.347E+08	5.221E+08	0.5586	8.457E+08
-66	4.485E+08	1.567E+08	0.3494	3.965E+08	1.356E+08	0.3421	4.058E+08	1.333E+08	0.3284	7.326E+08	3.839E+08	0.5240	6.489E+08
-64	3.868E+08	1.205E+08	0.3116	3.475E+08	1.085E+08	0.3124	3.539E+08	1.038E+08	0.2932	5.872E+08	2.745E+08	0.4674	5.297E+08
-62	3.389E+08	9.636E+07	0.2844	3.094E+08	8.681E+07	0.2806	3.124E+08	8.293E+07	0.2655	4.870E+08	2.009E+08	0.4125	4.426E+08
-60	3.030E+08	8.099E+07	0.2673	2.715E+08	7.087E+07	0.2610	2.814E+08	7.048E+07	0.2505	4.165E+08	1.502E+08	0.3606	3.731E+08
-58	2.709E+08	7.148E+07	0.2639	2.469E+08	6.289E+07	0.2547	2.544E+08	6.179E+07	0.2429	3.661E+08	1.157E+08	0.3161	3.320E+08
-56	2.453E+08	6.425E+07	0.2619	2.232E+08	5.806E+07	0.2601	2.309E+08	5.674E+07	0.2457	3.277E+08	9.359E+07	0.2856	2.995E+08

Freq. Sweep Tests (Bonus Information)



Summary and Conclusions

- When making the specimens for dual cantilever beam fixtures, one important consideration is uniform thickness – machining to a uniform thickness before cutting the specimens from a common slab can enhance reproducibility.
- The linear viscoelasticity regime can be discerned by comparing normalized storage moduli values over a range of deformations and finding the threshold at which the normalized value drops to 95% of the starting value.
- Clamping tension is not guaranteed when testing using frequency sweeps over a wide range of temperatures unless a procedure is used that ensures the clamps are tight at the coldest temperature.
- Long tests such as the frequency sweep sequences prescribed in this round robin may be problematic for the Q800 due to lack of sufficient reserve of liquid nitrogen in the gas cooling accessory.

Summary and Conclusions

- **Determination of T_g by locating the peak of the tan delta function was very repeatable and consistent with thermomechanical analysis results from another device.**
- **The β peaks varied with frequency but were repeatable for a given frequency. For the α peaks, there was no significant frequency effect.**
- **Of the three pertinent parameters (storage modulus, loss modulus, and tan delta), only the storage modulus showed variability issues, and that only took place on the "upper shelf" (below about -80 C).**


The End